National Bureau of Standards

TECHNICAL NEWS BULLETIN

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Diamond-Die Drilling Method Revolutionizes Industry

A new, electrical method for drilling small diamond dies, used in drawing and shaping extremely hard and fine wire, has been developed at the National Bureau of Standards, eliminating almost 100 man-hours from older processes. This discovery has completely revolutionized the fine-wire diamond-die industry, introducing it into the United States for the first time in history.

Although dies of tungsten, molybdenum, or boron carbide have in recent years competed with diamond dies, the diamond is still the only tool used in shaping the harder and tougher materials, such as chrome nickel, brass and phosphor bronze, and high-carbon steel wires. Moreover, wires finer than 15 ten-thousandths of an inch in diameter can be drawn only by diamond dies. In addition, the diamond is indispensable whenever precision of size and perfect roundness are essential considerations. Important uses for diamond-drawn wire are in radar and radio equipment, in high-frequency coil windings, as filaments for small tungsten lamps, and in delicate and precise electrical instruments. Wires of this type have become increasingly important with the expansion of the electronic and allied industries.

Prior to 1940, the indispensable small diamond dies were imported from Europe because labor costs made their manufacture unprofitable in this country. During the war, with all supplies from Europe cut off, the Bureau undertook an investigation to speed up the manufacturing process and improve the quality of dies to meet the emergency requirements for many vital war products.

A diamond die consists of five surface areas: the primary cone, the secondary cone, the reduction cone, the bearing, and the exit cone. In appearance such a die is similar to a wine glass. The portion of the die where the wire enters, the primary cone, corresponds to the goblet part of a wine glass. The narrowing stem from the bottom of the goblet to the base of the glass corresponds to the secondary cone, the reduction cone, and the bearing. Actual wire shaping takes place at the bearing. Finally, the base of the wine glass represents the exit cone.

Electric-Drilling Technics

The new process, developed by C. G. Peters, Chief of the Interferometry Section, and his associates, W. B. Emerson, K. F. Nefflen, F. K. Harris, and I. L. Cooter, replaces the older mechanical die cutting with high-voltage drilling, followed by electrolytic drilling.

The primary cone is drilled by means of high-voltage sparks formed at the point of a needle electrode in contact with the face of the diamond. These sparks release the energy stored in a condenser that has been charged to a high voltage by a transformer. The condenser is charged through a "quenched" gap consisting of a number of very small gaps in series.

When rising voltage is applied to the circuit, sparking

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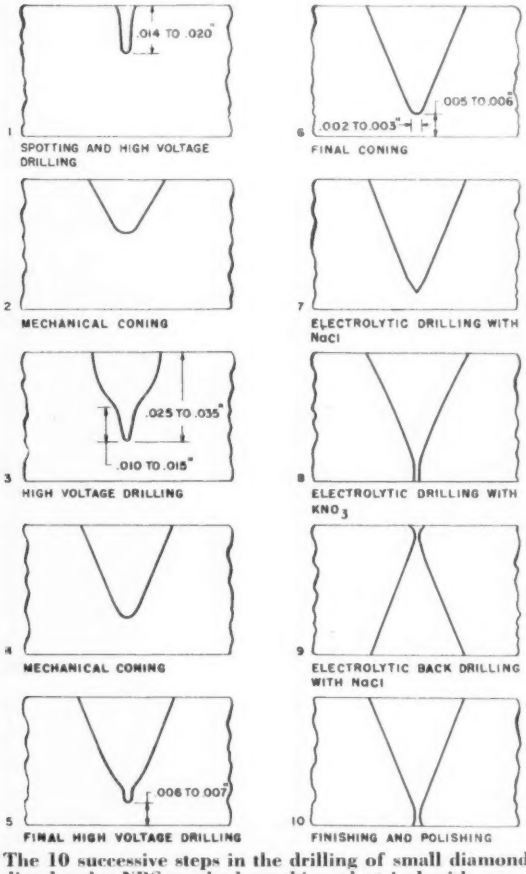
occurs first at the quenched gap. This is followed, as the voltage is increased, by a discharge across the face of the diamond between the needle electrode and the brass block on which the diamond is mounted. Without the quenched gaps in the circuit, the discharge would be a more or less continuous arc, overheating the diamond and producing a dark deposit on its surface. The rate of drilling increases with the power input into the circuit until a limit is reached where the temperature of the diamond is too high and its surface takes on a frosty appearance. The drilling needle, which is 0.02 inch in diameter, becomes red hot at its tip before this "frosting" point is reached, and the reddening point of the electrode is used as a criterion in adjusting the power input to the circuit.

The secondary cone is formed by the action of a low-voltage spark in an electrolytic solution. The diamond is mounted on an insulating post in a shallow glass dish, and enough of the electrolyte is used to just fill the dish and just cover the diamond. The "drilling" electrode is a platinum-iridium needle that is lowered into contact with the bottom of the primary cone and rests with very light pressure (around one-fifth of a gram) on the diamond. A second electrode dips into the solution at some distance from the diamond.



The 10 successive steps in the drilling of small diamond dies by the NBS method combine electrical with some mechanical operations. The method has reduced the average time for producing a diamond die by almost 100 hours. The simple drilling technic makes the work less tedious and more practical for both small- and large-scale enterprise.

When a low voltage (around 90 volts) is applied between the electrodes, sparking occurs at the tip of the drilling needle, and a smooth-walled conical hole is formed in the diamond directly under the needle. The shape of the hole and the angle of its walls are controlled by the type of electrolyte used, whereas the hole size is controlled by the pressure of the needle against the diamond. The power input into the circuit is controlled by the depth of the solution covering the diamond and by the voltage applied.

Finished Die in Ten Steps

The National Bureau of Standards method of making small diamond dies consists essentially of 10 steps, which combine electrical with some mechanical drilling. The die blank that is used in these operations has two plane-parallel faces between which the die will be formed, and a side window for viewing the progress of the drilling. These are cut on the diamond, using a rotating cast-iron lap with diamond powder as an abrasive. The cutting action of the lap is accelerated by the application of a high-voltage electric arc across the face of the diamond being cut.

In the first operation, 10 minutes of high-voltage drilling of the blank, called spotting, produces a cone

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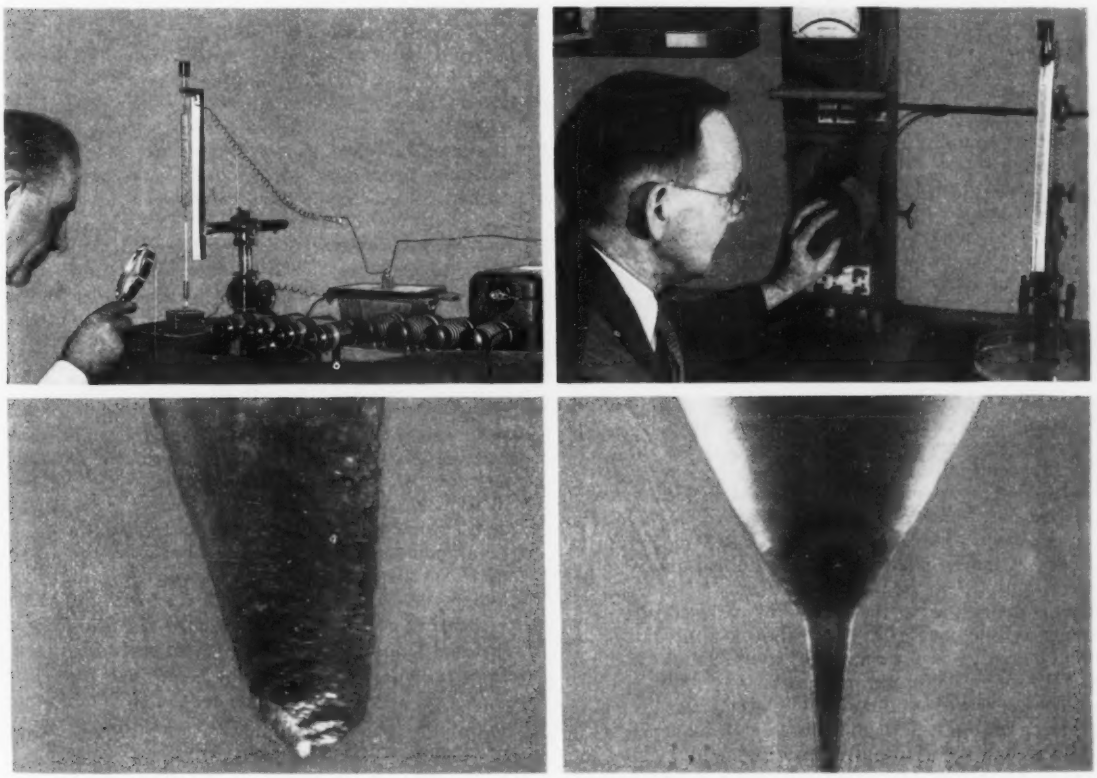
0.014 to 0.020 inch deep that serves as a pilot hole for the next operation. This is followed by 30 to 40 minutes of mechanical drilling to cone out the hole, using diamond powder in a gelatin solution as the abrasive. Operation three is repetition of the first high-voltage drilling and is followed by step four, mechanical coning. The final fore-drilling, operation five, should bring the bottom of the cone to between 0.006 and 0.008 inch from the back surface of the diamond. For exceptionally thick blanks, additional drilling and coning may be required. Step six, the final primary coning, demands greater care than the other similar operations to assure a symmetrical shape, free from drilling rings, and with either a polished or fine matt surface. The diameter of the cone immediately above its apex should be between 0.002 and 0.003 inch for good blending of the primary and secondary cones.

Operation seven begins the series of three electrolytic drilling operations, using two different solutions—sodium chloride and potassium nitrate. The first drilling of the secondary cone utilizes a 5-percent aqueous solution of sodium chloride.

Although an electrolyte of potassium nitrate produces a relatively long, narrow cone of correct size and shape for the secondary cone, the hole drilled, using this solution, tends to be out of alinement with the primary cone if drilling is attempted directly in a primary cone with a broad, unpointed, or irregular bottom. Excellent alinement and better blending of the primary and secondary cone are obtained by using sodium chloride, although an additional 30 minutes is needed for this step.

Drilling the main body of the secondary cone with potassium nitrate is similar to operation seven, except that the electrolyte solution is stronger and the tip diameter of the drilling electrode is smaller, ranging from 0.0010 to 0.0015 inch. After 40 to 45 minutes of drilling time, the drill will have pierced the back face of the die, giving a smooth-bore cone with a length of about 0.006 inch and a diameter at the bottom of 0.0005 to 0.0006 inch. If the initial distance between the bottom of the primary cone and the back of the die was greater than 0.005 inch, the drilling may not pierce the back of the diamond and an addi-



First step in the production of a diamond die (greatly magnified, lower right) by the NBS method is drilling of the primary cone, the cup-like portion of the die. High-voltage sparks formed at the point of a needle electrode in contact with the face of the diamond (upper left) produce the rough primary cone (lower left). This cone is completed by mechanical reaming. Drilling of the secondary cone is accomplished by the action of a low-voltage spark in an electrolytic solution. The drilling electrode, supported by a coil spring, rests lightly on the diamond, which is just covered by the solution of electrolyte (upper right). The die is completed by electrolytic drilling of a slight countersink (not shown) at the back surface to prevent chipping and undue wear.

tional 40 minutes of drilling with potassium nitrate, using a resharpened electrode, is necessary.

The sharp edge formed at the intersection of the secondary cone and the back surface is given a slight countersink in operation nine to prevent damage when wire is drawn through the die. This countersink is made by inverting the die beneath the drilling electrode and drilling electrolytically from the back for 4 or 5 minutes, using the solution of sodium chloride.

The final step is finishing and polishing, a mechanical operation that improves the blend between the primary and secondary cones, improves the roundness of the die, and improves the shape and polish of its working parts.

Improved Dies Through Electric Drilling

Electric drilling, producing superior dies, takes 7 to 15 hours, depending on the type of die, with 10 hours as an average for this new process. Actual man-hours are considerably less than this, averaging about 2 man-hours per die, because almost every stage in the process is easily adaptable to multiple production.

Dies produced by the NBS method have better wearing characteristics in service than those produced by purely mechanical methods. In mechanically drilled dies, holes are started in the top and bottom of the diamond by "bruting," or gouging, the face of the diamond with a small diamond chip. The hole is drilled through the body of the diamond by "pecking" it with a spinning steel needle charged with diamond powder, a sort of "star-drilling" process. These operations result in severe strains and minute fractures in the walls of the die cones and, although the magnitude of the strain resulting from mechanical drilling decreases with reduced load on the drilling needle and with the size of the diamond powder used, it is still present in most mechanically drilled secondary cones. Frequent failure by chipping at the intersection of the secondary and exit cones of dies so drilled is ascribed to weakening of the material by the mechanical treatment. Likewise, rapid wear of some dies at their initial size may be attributed to disruption of material, underlying the working surfaces of the dies, that has not been removed by sufficient polishing.

In contrast to the condition found in mechanically drilled dies, cones produced by either the high-voltage or the electrolytic drilling process show no strain in the surrounding material when examined under polarized light. The freedom from strain and fractures results in less wear as the die is used to draw wires. The electrical methods of die formation are therefore particularly adapted for small dies used in drawing wires 0.0015 inch in diameter, or smaller.

The electric method requires no special skills or long training on the part of operators. Equipment is inexpensive and the drilling technic is simple, making the work less tedious and more practical for small as well as large-scale enterprise. A satisfying development of the National Bureau of Standards investigation is that domestic die makers now find it advantageous to produce these small dies that formerly were obtained only through importation.

Standards for Analytical Filter Papers

Standards of quality for analytical filter papers, previously nonexistent for this important item in chemical research and analysis, are being developed by the Bureau's paper laboratory through a critical evaluation of all currently available brands. Based on improved testing methods perfected at the Bureau, together with established procedure for certain basic determinations, the standards as finally recommended will, it is believed, provide a ready means of determining the suitability of a particular paper for its intended use. Requirements will be included for the three classes of general analytical work involving coarse, medium, and fine precipitates.

Analytical filter papers, like many other laboratory materials, have an importance to science and industry far in excess of their dollar value. Many industrial processes require quick, accurate analyses for their control, and these depend, in many cases, on the speed, retentiveness, and purity of the filter paper used in making the analyses.

Filter papers have been used and bought by brand names practically as a tradition among laboratory workers, and preference has usually been given to imported papers, most of which have come from England, Germany, and Sweden. When the war made supplies from these sources uncertain, American manufacturers began the production of grades of analytical papers that had not previously been made in this country. In order to provide data that would be useful as a guide to manufacturers and users in comparing the quality of different papers, as well as to eliminate dependence on brand names, the Bureau undertook an investigation of the physical and chemical properties of the papers.

Improved methods have been devised for determination of rate of flow of water through the papers, retention of fine precipitates, and ash content, as well as a method for measuring the bursting strength of wet paper. Density, purity of cellulose, and acidity, for which definite testing procedures were already available, are also important determinations.

The method for rate of flow of water is of special interest. Unique in that it does not require the use of special, complicated apparatus, it possesses the advantage of simulating the manner in which filtrations are made. The filter paper is folded in a cone in the usual way, taking care to expel air pockets. Instead of placing it in a funnel, however, the cone is suspended freely in a wire loop, thus eliminating the variables such as design, angle, and stem dimensions of the funnel. When the water used in the test was prefiltered, highly reproducible results were obtained.

This work has shown significant differences among filter papers, which, if studied critically, may point the way to future improvements. For example, some papers of equal retentiveness differ in speed, and hence may yield a clue as to how still faster papers may be made.

Measurements of the quality of three foreign and two domestic brands of analytical filter paper have shown that the American made papers are fully equal to the imported papers. It is anticipated that the application of quality standards based on quantitative measurements will eliminate traditional buying practices and permit free competition among all suppliers.

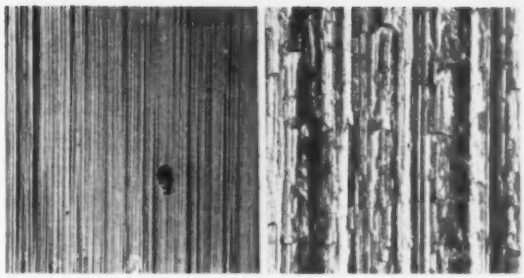
Replica Surface Analyzer

Evaluation of surface finish or roughness, essential in specifications for mechanically finished surfaces, is obtained by a new Bureau method in which a carefully prepared plastic replica of the surface is analyzed by photoelectric means.

The replica surface analyzer, developed by Harry K. Herschman of the Metallurgy Division, is based on the use of a plastic replica of a surface, which reproduces in minute detail the protuberances and recesses of the surface. The plastic replica is produced by applying a suitable solvent, or solution of plastic in solvent, to the test surface and pressing on a strip of clear plastic film. When dry, the film, or replica, is readily stripped from the surface.

The evaluation of a surface consists essentially in passing a restricted beam of light through an oscillating replica, thence through a suitably restricting diaphragm onto a photoelectric cell. The replica is oscillated with respect to the light beam in a straight line or in a curved path with the path of motion maintained so that the light beam will cross the protuberances and recesses of the surface.

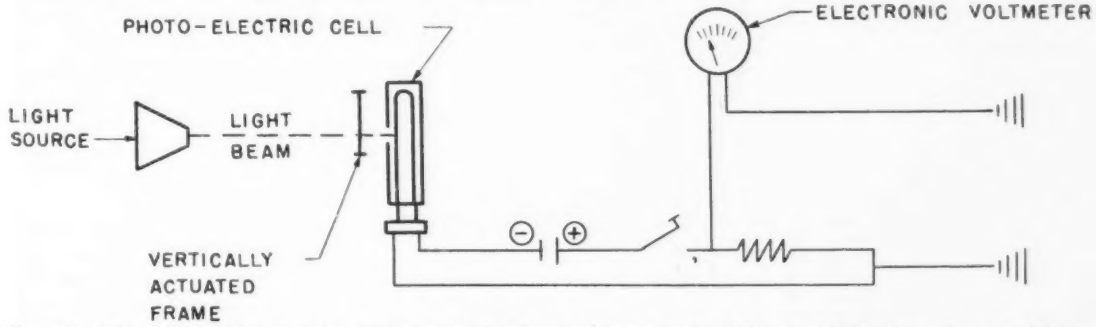
Even very minute variations in the number and nature of the protuberances and recesses on the replica film cause corresponding variations in the intensity and angle of refraction of the light transmitted by the replica. The light passing through the restricting diaphragm strikes the photoelectric cell and produces a correspondingly varying current. The average voltage variation, measured by a suitable alternating-current electronic voltmeter, is proportional to the variation—from increment to increment on the replica—of the amount of light allowed by the geometric form of each increment to reach the photoelectric cell. Meter readings, or records, are translated into "roughness" evaluations by calibrating against surfaces of known roughness as determined by microscopic means.



Micrographs of surfaces—finely ground (left) and coarse-shaped (right)—obtained by the use of plastic replicas employed in the replica method of evaluating surface roughness. Photographed with transmitted light at magnification of 100.

Some of the commonly used devices for determining the roughness of a surface employ a stylus, or tracer, that generally terminates in a spherical tip of very small radius (0.005 inch or less). These are usually made of a very hard material, such as diamond, which, in some cases during the tracing operation, may score the surface. This is especially true of materials of low hardness value, such as annealed steel or bearing alloys. Furthermore, this type of instrument measures only a very restricted area in a single setting. The method for evaluating roughness developed at the Bureau entails neither of the objectionable features of the stylus, or tracer devices.

The salient features of the replica method for surface analysis are (1) maintenance of a permanent record of a surface finish, (2) rapid evaluation of a relatively large area of surface in one determination, (3) simplicity of operation, (4) prevention of damage to surface, even for soft materials, such as lead or tin-base alloys, and (5) availability of the method, as the replica may be prepared in one locality and transported to the location of the analyzer. There are indications that this method may also be applicable for evaluating the corrosion pitting of metals.



The principle of the replica surface analyzer consists in passing a restricted beam of light through an oscillating test replica (mounted on vertical frame) onto a photoelectric cell. Light transmitted by the replica varies with the geometric characteristics of the replica and causes a fluctuation of electron photoemission and hence a pulsating current, which is registered on the alternating-current voltmeter. "Roughness" may be evaluated through a calibration of the voltage readings.

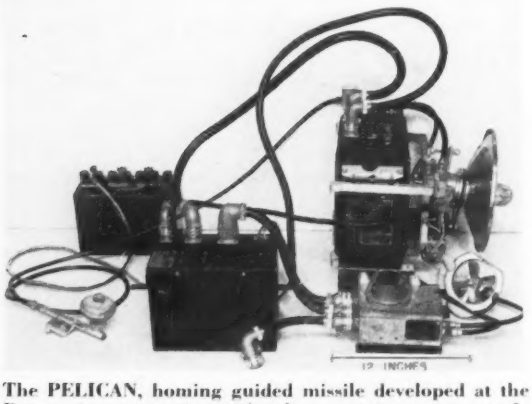
Guided Missiles

The BAT, first fully automatic guided missile successfully used in combat by any nation, offers evidence that guided missiles have passed from the experimental stage into practical application as a combat weapon. One of several guided missiles developed by the National Bureau of Standards under the sponsorship of the Navy Bureau of Ordnance, the BAT has led to further research on advanced forms, the "Kingfisher Series."

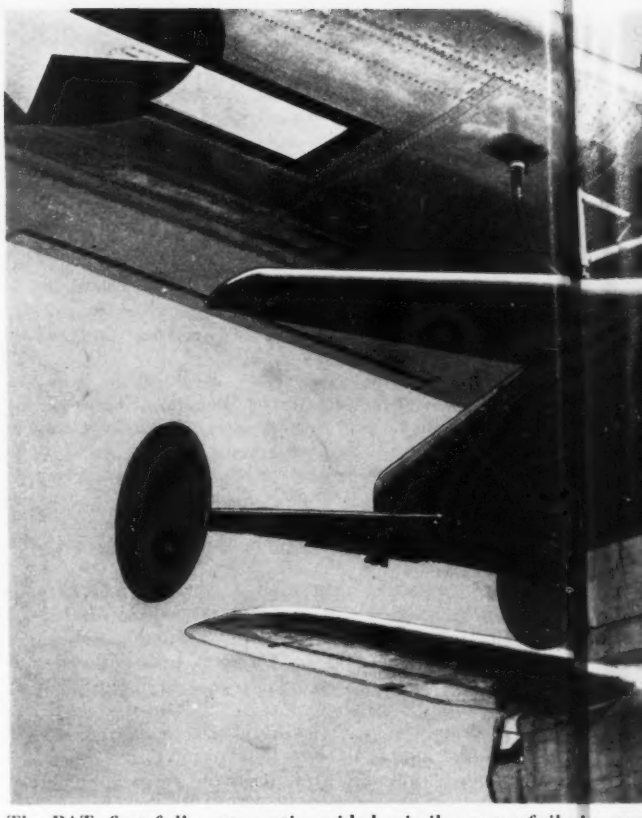
Work on guided missiles constituted one of the major war activities of the National Bureau of Standards. During the course of the work, such missiles as the ROBIN, the PELICAN, and the BAT were brought to a state of completed development. Because of the much greater operational flexibility of the BAT, however, it was the only one of these missiles carried into large-scale production and combat use. The program on guided missiles began in the fall of 1940, when the National Defense Research Committee requested the Bureau to permit the appointment of Dr. Hugh L. Dryden as aerodynamics consultant in connection with a contract for the development of a glide bomb capable of being directed to the target by radio remote control, utilizing television sighting.

ROBIN and PELICAN

In April 1942, the Bureau acquired the use of an area for flight tests near Warren Grove, N. J., where tests were made with aircraft supplied by the Navy. From this period on, the several projects designated ROBIN, PELICAN, and BAT were prosecuted simultaneously. Each of these projects utilized a glider in three sizes, designed to be carried externally by the dropping airplane. The unique type of aerodynamic control, suitable for homing missiles, was developed in the Bureau's Aerodynamics Laboratory.



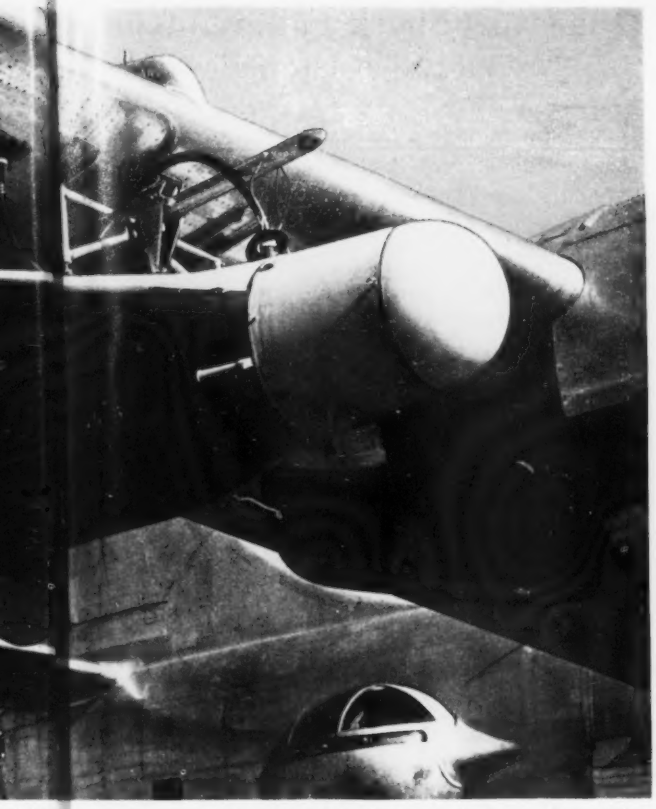
The PELICAN, homing guided missile developed at the Bureau in cooperation with other agencies, concurrently with the BAT, is directed by radar. The PELICAN, however, carries only receiving equipment (above), the transmitter being located on the "mother" plane.



The BAT, first fully automatic guided missile successfuily in com radar. Emitting pulsed microwave electromagnetic radlatine BAT ment also includes a gyroscopic stabilizing unit and a servoem to n was very effective against the Japanese during the last yeare war.

Project ROBIN contemplated a missile incorporating a television transmitter with pickup tube in the nose of the bomb and manual remote radio control by an operator with a television receiver in front of him. The full-scale missile was to carry a standard M–34 2,000-pound bomb as pay load. Twenty-six model gliders and fourteen full-scale gliders were flight-tested in connection with this project. Twenty-four of the flights were with television equipment. Gyrostabilization was introduced in the seventh test. The tests were concluded in July 1943, when radar homing appeared to be a more practical solution of the guided missile problem.

The PELICAN was a radar device, but the missile contained only a receiver; the transmitter was on the "mother" airplane. A pulsed radar system was used, the transmitter emitting short pulses of high intensity. Directional information obtained from the returning echoes activated mechanisms within the missile, which kept it headed for the target. The first flight test of a missile carrying an experimental radar receiver was



sfully in combat, is equipped with both transmitting and receiving radiation BAT is directed toward the target by radar echoes. Equip- a servoem to move the control surfaces of the air stabilizer. The BAT t year e war.

made at the Warren Grove test area on November 12, 1942, followed a month later by the first flight showing homing control, the transmitter in both cases being located on the ground.

Various difficulties, both aerodynamic and radar, as well as development of an improved target selector circuit, delayed final selection of a production design. The first production glider was tested against an elevated beacon on June 10, 1943. Attention was then turned to tests against a water target and to illuminating technics with the transmitter in an airplane, all previous tests having been made with transmitter on the ground. Tests of this nature were made against a barge in the Chesapeake Bay in October 1943. In 1944, however, the PELICAN was returned to an experimental status, largely because the BAT appeared to be nearly ready for use.

The BAT

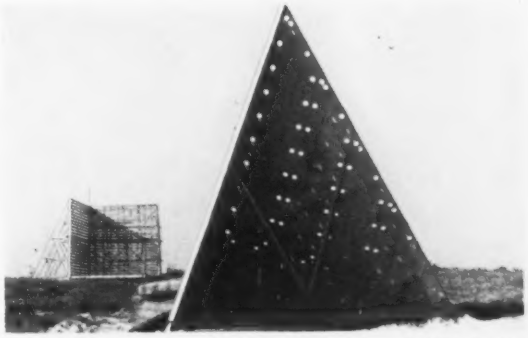
Designed to glide silently at the speed of a fighter plane, with a 1,000-pound bomb as cargo, the BAT is carried under the wing or fuselage of a Navy fighter or bomber and may be released at a distance as great as 14 miles from the target. Once the crew of the mother plane has located a ship or other target, the missile is set for this particular target and the release button is pushed. From then on the BAT automatically follows every maneuver of the target until it eventually strikes home, while the mother plane is free to proceed as its pilot wishes.

The code name BAT suggests the principle upon which this guided missile operates. A live bat gives out short pulses of sound and is guided by the echo, thus avoiding collision in the dark. The BAT missile emits pulsed microwave electromagnetic radiation and is directed by the radar echoes from the target. The radar robot pilot can, of course, "see" the target under any conditions of visibility.

In normal usage the target is first located by the standard search radar of the mother plane. The plane is then headed toward the target, and the radar transmitter and receiver in the BAT are aimed in its direction. Target data from the radar in the missile is displayed on a separate indicator in the mother plane under the control of the operator. After the radar equipment is manually adjusted to the proper conditions, it is switched to automatic tracking, and the missile is released. Echoes from the target are continuously detected by the radar receiver in the missile, and the output supplies corrective signals to the flight control units so as to direct the missile toward the target.

The outstanding features of the BAT as a combat weapon are (1) long range, which provides the ability to attack heavily armed targets with accuracy from outside the antiaircraft range; (2) improved accuracy, a result of the self-homing feature; (3) low angle of flight, which reduces high-altitude problems; (4) high pay load; and (5) self-guidance after release, permitting the carrying plane to maneuver as desired.

The BAT is equipped with a gyroscopic stabilizing unit and a servo-system to move the control surfaces of

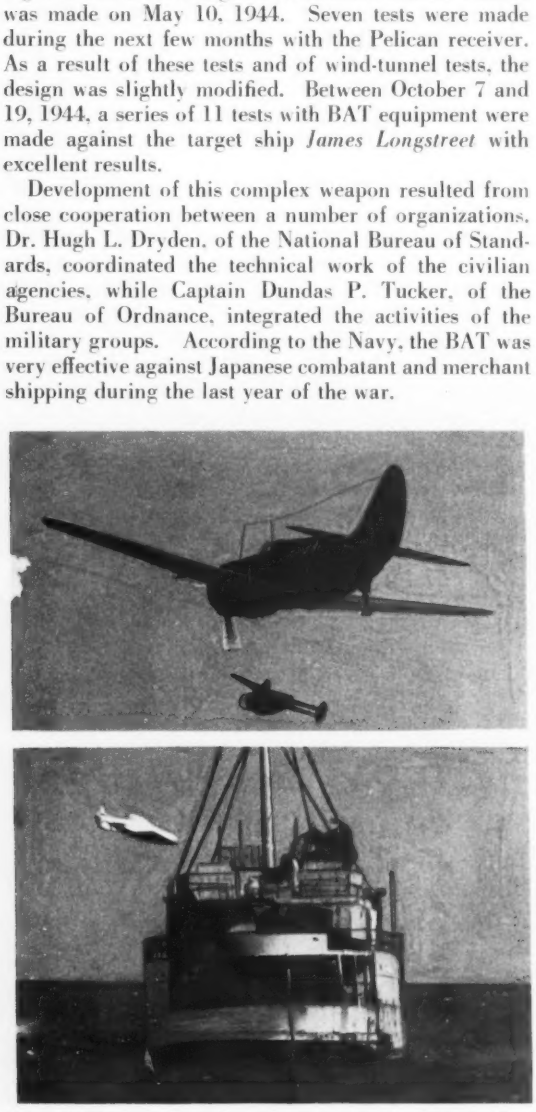


Corner reflectors used as targets in test flights of the BAT consist of three wire-covered planes intersecting at right angles. A radar signal striking any side is reflected back toward its source. The structure in the background was an earlier target model.

the air stabilizer. This equipment, together with the bomb load, is mounted in a glider-type airframe about 12 feet long with a 10-foot wing span.

The preliminary design of a glider of 10-foot span was started in June 1943, and construction of the first glider began approximately 3 months later. The Navy had placed a contract with Bell Telephone Laboratories for the design and development of the radar homing system early that year. In October these designs were crystallized, and in January 1944 one of the first three 10-foot gliders was sent to Bell Laboratories. The first flight test of a 10-foot glider with radio remote control was made on May 10, 1944. Seven tests were made during the next few months with the Pelican receiver. As a result of these tests and of wind-tunnel tests, the design was slightly modified. Between October 7 and 19, 1944, a series of 11 tests with BAT equipment were made against the target ship *James Longstreet* with excellent results.

Development of this complex weapon resulted from close cooperation between a number of organizations. Dr. Hugh L. Dryden, of the National Bureau of Standards, coordinated the technical work of the civilian agencies, while Captain Dundas P. Tucker, of the Bureau of Ordnance, integrated the activities of the military groups. According to the Navy, the BAT was very effective against Japanese combatant and merchant shipping during the last year of the war.



The BAT may be released from the "mother" plane (upper) at a range as great as 14 miles, and is automatically guided to the target (lower) by radar echoes.

Thermal-Shock Failure of Enamelware Utensils

A series of tests recently completed at the Bureau has provided an indication of the mechanism causing enamel to chip off cooking utensils when subjected to stresses due to sudden heating and cooling. The work, which supplements investigations made by the enamelware industry over a period of years, was conducted by W. N. Harrison and J. C. Richmond of the Enameled Metals Section, at the request of the Procurement Division of the Treasury Department.

Thermal-shock failure, or chipping, of an enamelware pan, occurs characteristically after the pan has been allowed to boil dry and cold water has been poured into it to counteract the excessive heat. This brings about severe shock, which, if repeated often enough, may cause failure of the enamel, and unless the pan is of good quality, may do so quickly.

In one group of experiments, using two identical sets of 2-quart pudding pans, the vessels were heated first, then one set was quenched quickly with water and the other allowed to cool before water was poured in. In the first case, the pans failed at an average of 7 cycles, that is after quenching seven times from successively higher temperatures, but in the second instance none of them chipped in 14 cycles, which was as far as the test was carried. These results demonstrated that the sudden quenching is the part of the cycle that starts the failure, although it may not show up before reheating. The explanation, given by Bureau investigators, is that quenching makes the surface contract faster than the underlying material, causing cracks that may be microscopic. The quenching water enters these cracks and, when the pan is reheated, forms steam that blows the enamel off.

Other experiments confirmed previous findings that a thin coat of enamel stands thermal shock better than a thick one. No consistent effect due to differences in thickness of the metal base was noted.

In the early test, a hot plate was used, and satisfactory results were obtained when 2-quart pudding pans were tested. It was thought desirable, however, to use a method that would heat the largest as well as the smallest utensils uniformly, so a thermostatically controlled gas-fired oven was developed in which the utensils were heated to predetermined temperatures. Ice water was then poured into them while they were still in the oven. This procedure was repeated at successively higher temperatures until a failure occurred, or until a stated series of quenchings had been completed without damage.

Specimens for the work were furnished by the Enameled Utensil Manufacturers Council. It is expected that these experiments, by contributing to a better understanding of the mechanism that causes thermal-shock failure, will be of assistance in the production and procurement of improved porcelain enameled utensils.

Countercurrent Reflux Still for Isotope Separation

A new and highly efficient type of countercurrent reflux molecular still for the separation of natural isotopes [1] has resulted from war-time research at the National Bureau of Standards with the cooperation of the United States Department of Agriculture. During the course of the work, several modifications of the apparatus have been developed in connection with research on mercury isotopes.

The discovery of practical means for obtaining isotopes furnished the chemist and biologist with a powerful tool—known as the method of tracers—for the study of chemical processes, particularly those which take place within the living organism. The method of tracers uses an isotope of a given element as a "tagged atom", which may be traced through a series of chemical reactions without loss of identity. Now that quantities of previously rare artificial radioactive isotopes are available, because of recent discoveries in nuclear physics, the progress in their utilization as tracers can be greatly expedited. However, for many applications of this method the stable, naturally occurring isotope of a given element is required. The countercurrent reflux still has proved to be an effective instrument for the separation of the natural isotopes of mercury, and is expected to have application to isotope work on other elements. Meanwhile, plans are being made for the application of this type of apparatus to the separation of pure hydrocarbons from petroleum and the isolation of vitamins from animal and vegetable products.

The term "molecular distillation" has been applied to that type of distillation where there is no return of escaping molecules to the evaporating surface. This is accomplished by operating with high-boiling (low-vapor-pressure) liquids under such high vacuum that the mean free path of the escaping molecules is of the order of the distance between the surface of the evaporating liquid and the cooled condensing surface of the still.

In molecular distillation, the relative rates of escape of the various types of molecules from a composite liquid surface are determined by two factors: (1) the vapor pressure (or the boiling point) of each component, and (2) the average molecular velocity of each component. Vapor-pressure differences among the isotopes of the heavy elements are small or nonexistent, whereas molecular velocities, at a given temperature, are inversely proportional to the square roots of the atomic weights. Molecular distillation thus offers a very practical means not only for separation of the isotopes of the heavier elements but also for the separation of any mixture of high-boiling substances differing in molecular or atomic weight. The latter application is particularly useful in the separation of hydrocarbons that do not differ appreciably in boiling point or are unable to withstand the high temperatures of ordinary distillation.

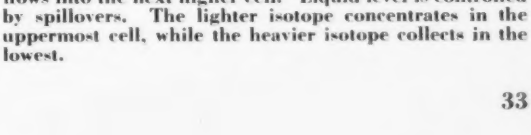
Previous work on the separation of mercury isotopes, using small single-stage molecular stills, has had little practical value because of the labor involved, the time consumed, and the large quantity of mercury required. In order to obtain appreciable concentration it is necessary to repeat the process many times. This requires an elaborate system for collecting, recombining, and distilling fractions.

To overcome these difficulties, a new type of molecular still was designed in which a number of single-stage stills are so connected that recombination of fractions takes place automatically by gravity feed. In this way, the necessity of making repeated "cuts", with consequent reduction in output, is eliminated. The resultant separation realized in one operation is equivalent to the separation per stage raised to the power of the number of stages. As a result, the time and labor involved become much less than for repeated distillation in a single-stage still. For example, a separation that would have required 55 individual and recombination distillations was obtained in one step with a 10-compartment still. In addition, the quantity of mercury required is enormously reduced.

The multistage molecular still consists of a series of evaporating surfaces, or pools, set adjacent to each other but at slightly different levels. A cooled roof directly above each pool serves to condense the vapor. The roofs are so sloped that the condensate will run along the surface to fall into the adjacent cell higher up. Each pool is equipped with a spillover, which allows liquid to run back in amount equal to the condensed vapor carried forward. The over-all operation is such that the light fraction increases in concentration toward the upper end, while the heavy fraction increases in concentration toward the lower end of the still.

In addition to the great savings of time, labor, and materials effected through use of this still, there are further advantages. The operation can be made continuous; the material to be concentrated can be fed into the system at one end or at the midpoint, if both light and heavy fractions are of interest; and the concentrate can be withdrawn continuously. Furthermore, the entire separation can be run without exposing the material to the atmosphere; this is particularly desirable for liquids that are susceptible to decomposition upon exposure to air.

[1] Details of this work are described in Bureau Research Paper RP1764, Concentration of the isotopes of mercury by free evaporation in a 10-cell countercurrent reflux still, A. Keith Brewer and Samuel L. Madorsky, J. Research NBS **38**, 129 (1947).



In the operation of a multistage molecular still, vapor from the liquid in a given cell condenses on the roof and flows into the next higher cell. Liquid level is controlled by spillovers. The lighter isotope concentrates in the uppermost cell, while the heavier isotope collects in the lowest.

Rotary Concentric-Tube Distilling Column

A new rotary concentric-tube distilling column, having a very high efficiency factor for fractional distillation, has been developed at the National Bureau of Standards by Charles B. Willingham, Vincent A. Sedlak, James W. Westhaver, and Frederick D. Rossini, in connection with the cooperative research program on hydrocarbons sponsored jointly by the Bureau and the American Petroleum Institute.

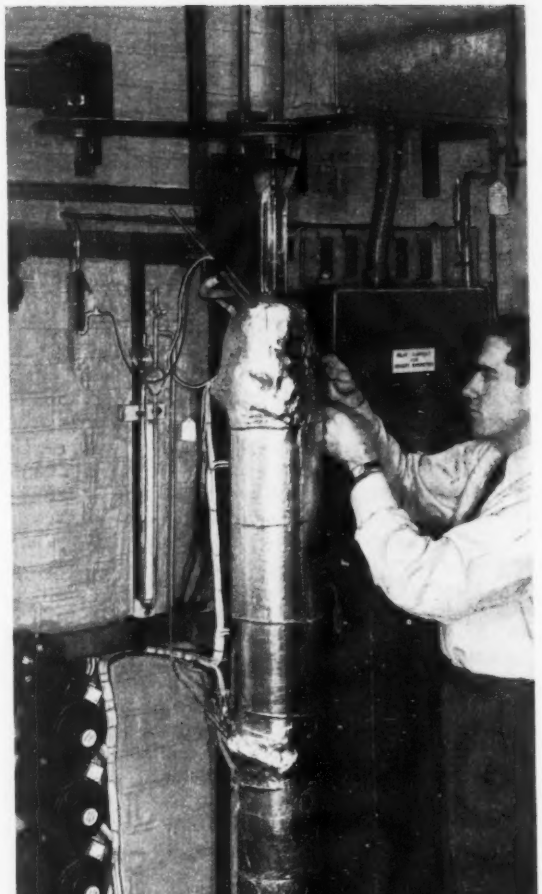
The present-day demand for fuels providing greater efficiency, higher speeds, and more concentrated power, as well as the development of various devices making use of new types of hydrocarbons, has resulted in a transition in the petroleum industry from the manufacture of broad fractions toward the production of narrower fractions and even pure compounds. The experimental program now under way at the National Bureau of Standards has two objectives: (1) the investigation of the chemical constituents of the crude oil based upon the actual isolation of pure hydrocarbons; (2) the securing of high-purity standard samples for the calibration of instruments used in analyzing such complex mixtures as aviation fuels and synthetic rubber components. The new distilling column, which is expected to have wide application to fractionation processes, will aid materially in the Bureau's hydrocarbon research program.

Numerous devices, based on both theory and experiment, are available for producing high separating efficiencies in distilling columns. Among these are open tubes of small diameter, concentric tubes with a small annular space, and parallel plates with a small space between them. All, however, are limited in use by a relatively low throughput, that is, quantity of material that may be volatilized per hour. According to theory, the efficiency may be improved by decreasing the spacing of the tubes or plates of the column, by decreasing the throughput, or by increasing the rate of diffusion of the gas molecules through the distilling column. Decrease in the spacing (or diameter) has already been carried about as far as practicable, while decreasing the throughput below the present low values is not feasible. Moreover, in a static apparatus, for a given temperature and composition, the rate of diffusion of the gaseous molecules is substantially constant.

Accordingly, the new distilling column was designed to improve separating efficiency by increasing the diffusion rate of the molecules in the gas phase. This is accomplished by forcing the gas into turbulence through rotation of the inner closed cylinder in a concentric-tube rectifying section.

The apparatus consists of three parts—the Pyrex glass head, steel rectifying section, and Pyrex pot. All three sections are provided with external heating elements and copper-constantan thermocouples for temperature regulation.

The steel rectifying section of this distilling column is the empty annular space, 0.043 inch (1.09 mm) wide,



The operator removes a test sample of distillate from the rotary concentric-tube distilling column. The rectifying section of the column is an empty annular space formed by the surface of a steel cylindrical rotor and the inside surface of the stationary outer wall.

formed by the inside surface of a stationary outer cylinder and the outside surface of a rotating closed inner cylinder, 2.928 inches (7.44 cm) in outside diameter and 23.0 inches (58.4 cm) in length. A motor-and-pulley system drives the rotor at speeds up to 4,000 rpm. The outer cylinder is enclosed in an asbestos-covered metal heating jacket surrounded by three separate Nichrome heating elements for the top, middle, and bottom portions of the jacket, respectively. The heating elements are covered externally with magnesia insulation and a layer of aluminum foil.

The Pyrex glass head consists principally of a water-jacketed condenser directly above, and opening into, an electrically heated chamber, in which the liquid reflux may be collected and sampled by means of a glass valve.

The heated chamber is surrounded by asbestos-wool insulation and aluminum foil.

The pot, made of a 3-inch Pyrex pipe, is sealed at one end and provided with a butyl carbitol manometer and a tube for withdrawing samples. Heated externally by a special sleeve-type heater, it is surrounded by magnesia insulation covered with aluminum foil.

Three thermoelements are provided. One measures the difference between the temperature of the top portion of the rectifying section and the liquid-vapor equilibrium in the head. Another measures the difference between the temperature of the middle portion and the mean temperature of the top and bottom portions of the rectifying section. A third is used to determine the difference between the temperature of the bottom of the rectifying section and the temperature of the liquid in the pot.

For high value of throughput—2 to 4 liters of liquid per hour—this distillation column, when operated at 4,000 rpm, has an efficiency factor about 10 times those previously reported for other rectifying columns. The efficiency factor changes relatively little with throughput at a given speed of rotation, but increases markedly with speed of rotation. This column also has low values of pressure drop per unit throughput, which may be quite advantageous for distillations at low pressure.

NBS Scientists

The President's Certificate of Merit has recently been awarded to **Dr. Hugh L. Dryden,** Associate Director of the Bureau, for his contribution to development of the BAT, the only automatic guided missile successfully used in combat. The award was presented to Dr. Dryden by W. John Kenney, Assistant Secretary of the Navy.

Dr. E. C. Crittenden, Associate Director of the Bureau, has been designated to carry added duties as chief of the Optics Division. **Lauriston S. Taylor,** head of the X-Ray Section, has been selected to act as Dr. Crittenden's assistant. Mr. Taylor will also continue as chief of his section. **Dr. Fred L. Mohler,** who has been acting chief of the Atomic Physics Section, has been named head of the Mass Spectrometry Section in the Division of Organic and Fibrous Materials.

Dr. John A. Hipple, one of the world's leading authorities on the design, construction, and use of the mass spectrometer, has been appointed chief of the Bureau's Atomic Physics Section. In this capacity, he will direct research on the ionization and separation of molecules by electron impact, and on the processes involved in these phenomena. The section will also embark on an extensive program of measuring the masses of the elements and their isotopes, using mass spectrometers to be designed by Dr. Hipple and constructed under his supervision.

Russell B. Wright, recently appointed as physicist and expert ordnance consultant to the chief of the Ordnance Development Division, will evaluate the over-all program of the Division both in regard to ordnance projects and to the civilian electronics program. He will also advise on the selection of special technical methods and represent the Division Chief on important committees and panels.

Frederick J. Bates, sugar physicist and chief of the Bureau's Optics Division, retired on January 31, after more than 43 years of continuous Bureau service, having joined the staff in 1903 as chief of the Polarimetry Section. An authority in the application of natural and magnetic polarization measurements to sugar tests, he devised the methods and prepared the Treasury Regulations for weighing, gaging, sampling, classifying, and testing imported sugars. Mr. Bates developed many instruments used widely in the physical and chemical testing of sugars, and has directed research on polarized light; formulation, growth, and properties of crystals; and the annealing of glass.

NBS Publications

Periodicals

Journal of Research of the National Bureau of Standards, Volume 38, Number 2, February 1947 (RP1765 to RP1772, inclusive), 30 cents.

Technical News Bulletin, volume 31, number 2, February 1947. 10 cents.

CRPL-D30. Basic Radio Propagation Predictions for May 1947 three months in advance. Issued February 1947. 15 cents.

Nonperiodical

RESEARCH PAPERS[2]

RP1758. The vibrational frequencies of semirigid molecules: a general method and values for ethylbenzene. William J. Taylor and Kenneth S. Pitzer. 15 cents.

RP1759. Analysis of a standard sample of natural gas. Martin Shepherd. 20 cents.

RP1760. Purification, purity, and freezing points of 8 nonanes, 11 alkylcyclopentanes, 6 alkylcyclohexanes, and 4 butylbenzenes of the API-Standard and API-NBS series. Anton J. Streiff, Evelyn T. Murphy, Janice C. Cahill, Helen F. Flanagan, Vincent A. Sedlak, Charles B. Willingham, and Frederick D. Rossini. 20 cents.

RP1761. Infrared absorption of some experimental glasses containing rare earth and other oxides. Ralph Stair and Conrad A. Faick. 10 cents.

RP1762. Properties of water-repellent fabrics. John W. Rowen and Domenick Gagliardi. 10 cents.

RP1763. Adsorption of water vapor by untanned hide and various leathers at 100° F. Joseph D. Kanagy. 10 cents.

RP1764. Concentration of the isotopes of mercury by free evaporation in a countercurrent reflux still. A. Keith Brewer and Samuel L. Madorsky. 10 cents.

[2] Send orders for publications under this heading only to the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Annual subscription rates: Journal of Research, $4.50 (foreign, $5.50); Technical News Bulletin, $1.00 (foreign, $1.35); Basic Radio Propagation Predictions, $1.50 (foreign, $2.00). Single-copy prices and prices of other publications are indicated in the lists.

[3] Reprints from January Journal of Research.

Miscellaneous [2]

M177. Tests of instruments for the determination, indication, or recording of the specific gravities of gases. Francis A. Smith, John H. Eiseman, and E. Carroll Creitz. $1.00.

Commercial Standard [2]

CS137–46. Size measurements for men's and boys' shorts—woven fabrics. 5 cents.

Simplified Practice Recommendations [2]

R9–47. Galvanized woven-wire fencing and barbed wire. (Supersedes R9-28.) 10 cents.
R100–47. Welded chain. (Supersedes R100–29.) 10 cents.
R223–47. Wire nails and staples. 10 cents.

Handbooks

(Amendments and Corrections)

Amendments to the model state law on weights and measures adopted by the Thirty-Second National Conference on Weights and Measures. (To accompany H26, Weights and Measures Administration.) Free upon request from the National Bureau of Standards, Washington 25, D. C.

Correction sheets, changes adopted by the Thirty-Second National Conference on Weights and Measures. (To supplement H29, Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices.) Free upon request from the National Bureau of Standards, Washington 25, D. C.

Letter Circulars [1]

LC845. Weights and measures—a selected list of publications of the National Bureau of Standards. (Supersedes LC772.)
LC846. Electric batteries and standard cells: Publications by the staff of the National Bureau of Standards, and references to other sources of information. (Supersedes LC720.)

Articles by Bureau Staff Members in Outside Publications [3]

Comparative bond efficiency of deformed concrete reinforcing bars. Arthur P. Clark. Journal of the American Concrete Institute (7400 Second Boulevard, Detroit 2, Michigan). **18**, No. 4, 381 (December 1946).

The problem of building code improvement. George N. Thompson. Law and Contemporary Problems (Duke University, Durham, N. C.) **12**, No. 1, 95 (Winter 1947).

Scientific evidence in crime detection. Wilmer Souder. Proceedings of the International Association for Identification, 1946 (Leroy Goodwin, Secretary, Youngstown, Ohio).

Caucho natural y caucho sintetico. Norman Bekkedahl. Revista Facultad Nacional de Agronomia (Facultad Nacional de Agronomia, Universidad Nacional, Medellin, Colombia) **6**, No. 22, 53 (June 1946).

[1] Available on request from the National Bureau of Standards, Washington 25, D. C. Letter Circulars are prepared to answer specific inquiries addressed to the Bureau, and are sent only on request to persons having a definite need for the information. The Bureau cannot undertake to supply lists or complete sets of Letter Circulars or send copies automatically as issued.

[3] These publications are not available from the Government. Requests should be sent direct to the publishers.

Periodicals of the National Bureau of Standards

The National Bureau of Standards is the principal agency of the Federal Government for fundamental research in physics, chemistry, and engineering. Some 2,300 individuals comprise the staff of the Bureau. The scientific and technical activities are divided into 11 divisions: electricity, metrology, heat and power, optics, chemistry, mechanics and sound, organic and fibrous materials, metallurgy, mineral products, ordnance development (including electronics), and radio propagation. The results of research, development, and test at the National Bureau of Standards in these fields of physics, chemistry, and engineering are regularly reported in 3 monthly publications: the Technical News Bulletin, the Journal of Research, and the Basic Radio Ppopagation Predictions.

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Technical News Bulletin

A monthly report containing concise articles on current work in the NBS laboratories. The articles are brief, with emphasis on results and applications rather than on experimental details. Abstracts of papers appearing in the Journal of Research, accounts of important national and international scientific meetings, and bibliography of publications by staff members also appear here. The Bulletin is designed primarily to give a general and timely review of the Bureau's research.

Journal of Research

A monthly publication presenting regular papers reporting the research of the Bureau in physics, chemistry, and engineering. The Journal is, in content and format, similar to the classical scientific periodicals.

Basic Radio Propagation Predictions

Each monthly issue presents complete radio propagation information, enabling the reader to calculate the best sky-wave operating frequencies over any path at any time for average conditions for the month of prediction. Predictions are issued 3 months in advance.

U. S. GOVERNMENT PRINTING OFFICE: 1947